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IS 12042 (1987): Method for determination of molybdenum by atomic absorption spectrophotometer [CHD 1: Inorganic Chemicals]



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Indian Standard

METHOD FOR
DETERMINATION OF MOLYBDENUM BY
ATOMIC ABSORPTION SPECTROPHOTOMETER

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHOD FOR DETERMINATION OF MOLYBDENUM BY ATOMIC ABSORPTION SPECTROPHOTOMETER

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Indian Standard

METHOD FOR DETERMINATION OF MOLYBDENUM BY ATOMIC ABSORPTION SPECTROPHOTOMETER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 16 February 1987, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is based on the fact that atoms present in ground state will absorb incident light of the same wavelength they emit when excited. When radiation from the given excited element, normally hollow cathode lamp, is passed through the flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms present in flame. A hollow cathode lamp for the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the sample solution into the flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and photosensitive device measures the attenuated transmitted radiation. Various elements including molybdenum can be precisely analyzed by this technique.

0.3 Various products, where molybdenum estimation may be required, include metals and alloys, chemicals, ores and minerals, etc.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the method for the determination of molybdenum by atomic absorption spectrophotometer.

2. OUTLINE OF THE METHOD

2.1 The sample is brought into solution by suitable treatment with acid or acid combinations or by alkali fusion, diluted with distilled water,

*Rules for rounding off numerical values (*revised*).

filtered and suitable dilutions are made for aspiration into air-acetylene flame for better sensitivity into the nitrous oxide-acetylene flame. The standard solution is made in the same way for calibration. The most sensitive molybdenum line is 313.26 nm, however, other lines suitable for higher concentration can also be used.

3. INTERFERENCES

3.1 Calcium, iron, manganese, sulphate and strontium depresses the response in air-acetylene flame. The effects may be reduced by the addition of 0.5 percent $\frac{m}{m}$ of aluminium or 2 percent $\frac{m}{m}$ of ammonium chloride to both sample and calibration solutions or preferably by use of hotter flame. In the nitrous oxide-acetylene flame the response is slightly depressed in the presence of calcium and iron. The effect may be reduced by the addition of 0.5 percent $\frac{m}{m}$ of aluminium chloride or 1 percent $\frac{m}{m}$ of sodium sulphate to both sample and calibration solutions. The sensitivity for molybdenum drops when pressure in the acetylene cylinder becomes low. The cylinder should be changed when pressure falls below 7 kg/cm².

4. APPARATUS

4.1 Atomic absorption spectrophotometer having following parameters can be used for determination of molybdenum:

- a) Lamp current — depending on the lamp and instrument used.
- b) Fuel — Nitrous oxide-acetylene.

NOTE — Acetylene flow rate is critical and must be carefully adjusted to give a flame with maximum 'feather' height without luminiscence.

- c) Band pass — depending on the instrument used normally 0.15 to 0.20 nm.
- d) Wavelength and working range.

<i>Wavelength (nm)</i>	<i>Working Range (µg/ml)</i>
	<i>Nitrous oxide-acetylene flame</i>
313.3	1 to 10
320.9	10 to 100

4.2 Safety Precaution — Follow the manufacturer's recommendation on igniting and extinguishing the flame.

5. REAGENTS

5.1 Pure Molybdenum Powder — 99.99 percent.

5.2 Concentrated Nitric Acid — See IS : 264-1976*.

5.3 Concentrated Hydrochloric Acid — See IS : 265-1976†.

5.4 Perchloric Acid — 70 percent.

NOTE — Perchloric acid is a strong oxidizing agent. It will explode in contact with organic materials or by shock or heat and is strongly irritant.

5.5 Standard Molybdenum Solution — Dissolve 1.0 g of molybdenum powder in 10 ml of hydrochloric acid and 10 ml of deionized water. Add 1.0 ml of nitric acid. Dilute to 1 litre in a volumetric flask with deionized water to give 1 000 $\mu\text{g/ml}$ of molybdenum. Store in a polyethylene bottle.

5.6 Ionization Buffer — Dilute potassium chloride solution (1 mg/ml).

6. SAMPLE PREPARATION

6.1 Metals and Alloys — A suitable quantity of the sample is dissolved in 1:1 hydrochloric acid and perchloric acid (70 percent) and heated until dense fumes are evolved; over-heating should be avoided, otherwise molybdenum gets precipitated. Cool, dilute and filter. Make up to known volume. A suitable dilution is made for the determination of molybdenum before aspirating into the flame.

6.2 Chemicals — The sample is brought into solution by wet digestion with hydrochloric acid/perchloric acid. Filtrate is made up to suitable known volume.

6.3 Ores and Minerals — Finely divided particles dissolved in hydrochloric, perchloric and nitric acid mixture (2:2:1 ratio). Dissolve the mass in 30 ml of acid mixture, heated to fumes, cooled, diluted and filtered to remove silicious matter.

Residue is dried, ignited and fused with 0.5 g of borax. Extract in hydrochloric acid is taken and mixed with the original solution. The final solution is made up to volume.

NOTE — All dilutions to be made with ionization buffer.

7. PROCEDURE

7.1 Optimize the response of the instrument by adjustment of burner height, flame and monochrometer as directed in the operation manual of the instrument. Aspirate water to get zero absorbance, when stable

*Specification for nitric acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

response is observed, aspirate standard (at least four solutions) and note down absorbance. Aspirate sample to get absorbance of the sample. Prepare calibration curve by plotting the net absorbance value of the standard against concentration in $\mu\text{g/ml}$ of molybdenum. Locate the print of the sample absorbance and calculate the concentration of molybdenum in the sample.

8. CALCULATION

8.1 Molybdenum, percent by mass =
$$\frac{C \times V}{10^6} \times \frac{100}{M}$$

where

C = concentration of molybdenum in $\mu\text{g/ml}$ in final solution,

V = volume in ml of the final solution, and

M = mass in g of the sample in final solution.